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**Title of the Invention: Treatment Method for Liquid Crystal Polymer Molded Article**

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(54) **Title of the Invention:** **Treatment Method for Liquid Crystal Polymer Molded Article**

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## SPECIFICATION

### 1. Title of the Invention

**Treatment Method for Liquid Crystal Polymer Molded Article**

### 2. Claims

(1) A treatment method for a liquid crystal polymer molded article, characterized in that a molded article consisting of a liquid crystal polymer is subjected to a roll calender treatment.

(2) The treatment method for a liquid crystal polymer molded article according to Claim 1, wherein the liquid crystal polymer is a liquid crystal polyester consisting of a p-hydroxybenzoic acid component and a polyethylene terephthalate component.

(3) The treatment method for a liquid crystal polymer molded article according to Claim 1 or 2, wherein the molded article is a film, sheet or fiber.

## 3. Detailed Description of the Invention

### (Field of Industrial Utilization)

The present invention relates to a treatment method for a liquid crystal polymer molded article used to suppress fibrillation or delamination of this liquid crystal polymer molded article.

### (Prior Art)

Liquid crystal polymers (thermotropic liquid crystal polymers) can be melted and molded, are superior in terms of heat resistance and dimensional stability, and can yield molded articles that have a high strength, high elastic modulus, and high gas barrier properties. Accordingly, such polymers have attracted attention, and some molded articles consisting of liquid crystal polyesters have been adapted for practical use.

However, liquid crystal polyesters have conspicuous uniaxial orientability and multilayer formability, so that various problems arise when such polymers are molded into molded articles. For example, in cases where the T die method is used when films are formed, the strength in the longitudinal direction (MD direction) is large, but the strength in the lateral direction (TD direction) is conspicuously small. If the inflation method is used, a biaxially oriented film that tends not to rupture is obtained; however, the structure of this film is a multilayer structure, and peeling between layers tends to occur. Moreover, in cases where fibers are formed by melt spinning, a shear force is applied only in the discharge direction, so that the fibers are oriented to a high degree only in the axial direction. Consequently, the cohesive force between molecular chains is weak, and fibrillation easily occurs as a result of buckling or friction during working or use, thus causing a degradation of mechanical properties and the generation of nap.

A method in which films or fibers are melted and bonded by hot pressing is conceivable as a way of solving these problems; if such a method is used, however, the oriented state is destroyed, and the physical properties show a severe deterioration.

### (Problems to Be Solved by the Invention)

It is an object of the present invention to provide a treatment method for a liquid crystal polymer molded article which makes it possible to obtain molded articles that are free from

problems such as fibrillation and interlayer peeling without any loss of the superior characteristics of such liquid crystal polymer molded articles.

**(Means Used to Solve the Above-Mentioned Problems)**

The present inventors conducted diligent research in order to solve the abovementioned problems; as a result of this research, the inventors discovered that it is effective to use a roll calender treatment and to apply press bonding to liquid crystal polymer molded articles without melting. This discovery led to the perfection of the present invention.

Specifically, the main point of the present invention is a treatment method for a liquid crystal polymer molded article characterized in that a molded article consisting of a liquid crystal polymer is subjected to a roll calender treatment.

In the present invention, the term "liquid crystal polymer" refers to a polymer that has the property of allowing the passage of polarized light in a molten state in an optical system equipped with polarizers that cross each other at 90°.

Typical examples of such polymers include (1) polyesters consisting chiefly of p-hydroxybenzoic acid, (2) polyesters consisting chiefly of 2-hydroxy-6-naphthoic acid, (3) polyesters consisting chiefly of terephthalic acid and hydroquinones, (4) polyesters consisting of a p-hydroxybenzoic acid component and a polyethylene terephthalate component, or the like. Within limits that cause no loss of the physical properties of the liquid crystal polyester, such polymers may also appropriately contain aromatic components such as isophthalic acid, m-hydroxybenzoic acid, 4,4-dicarboxybiphenyl, 2,6-naphthalenedicarboxylic acid, 4,4-dicarboxydiphenyl ether, resorcin, hydroquinone, 2,6-dihydroxynaphthalene, 4,4-dihydroxybiphenyl, 2-phenylhydroquinone, 2,2-bis(4-hydroxyphenyl)propane, bis(4-hydroxyphenyl)sulfone, 2,2-bis(4-β-hydroxyethoxyphenyl)propane, bis(4-β-hydroxyethoxyphenyl)sulfone and the like; aliphatic components such as malonic acid, succinic acid, suberic acid, adipic acid, azelaic acid, sebacic acid, dodecanedioic acid, tetradecanedioic acid, eicosanedioic acid, trimethylene glycol, propylene glycol, 1,4-butanediol, 1,6-hexanediol, diethylene glycol, triethylene glycol, polyethylene glycols and the like; and alicyclic components such as 1,4-cyclohexanedicarboxylic acid, 1,4-cyclohexanedimethanol and the like, as

copolymerized components. Furthermore, such polymers may also contain additives such as flame retarding agents, heat-resistant agents, light-resistant agents, delustering agents, pigments and the like.

Especially desirable among the abovementioned polyesters are polyesters of type (4), and a polyester consisting of a p-hydroxybenzoic acid component in a ratio of 30 to 74 mol% and a polyethylene terephthalate component in a ratio of 70 to 26 mol% is [especially] suitable from the standpoint of superior molten moldability and workability. For example, this liquid crystal polyester is manufactured by a method in which polyethylene terephthalate and p-acetoxybenzoic acid are mixed in specified proportions, an acidolysis reaction is then performed by heating and melting this mixture, and polycondensation is performed under reduced pressure.

Films, sheets and fibers are typical examples of molded articles treated in the present invention; however, the method of the present invention can also be applied to injection-molded articles and the like.

In the case of films, a product showing a balanced degree of orientation in the MD direction and TD direction is desirable from a practical standpoint; here, a film in which the tensile strength ratio between the MD direction and TD direction is 5:1 to 1:3 (preferably 4:1 to 1:2) is desirable.

The following method exists as a desirable method for obtaining such a biaxially oriented film.

Specifically, this is a method in which a shear force is applied in the direction perpendicular to the discharge direction when a melt of the liquid crystal polymer is extruded from a slit. A method in which a film is manufactured while rotating either a die lip or an insert, or both, using an inflation molding apparatus with a structure that allows the rotation of such a die lip or insert (or both) can be desirably used as a method for applying a shear force in the direction perpendicular to the discharge direction.

Furthermore, a method in which a melt is discharged from a spinneret in the same manner as in the case of an ordinary polyester such as a polyethylene terephthalate is generally used as a

method for manufacturing fibers; however, a method in which a film, especially a biaxially oriented film, is manufactured, and this film is then slit, is also effective.

In the present invention, a roll calender treatment is applied to such a molded article. Systems such as a steel/steel system, steel/rubber system, rubber/rubber system or the like can be used as the roll calender in this case; however, a steel/steel system is most desirable. The rolls may be subjected to embossing, or grooves may be cut in the rolls.

The roll pressing method may use a spring system, mechanical system, oil pressure system, air system, electromagnetic system, water pressure system or the like, and a system that is capable of applying a high pressure is desirable. A pressure of 1 to 300 kg/cm (line pressure) is suitable as the treatment pressure.

Furthermore, a method such as a catalytic heating system, induction heating system or the like can be used as the roll heating method. It is necessary to set the roll heating temperature at a temperature that is suited to the pressing force; ordinarily, this temperature is set at 50 to 250°C.

Furthermore, in the case of a film or sheet, the molded article is ordinarily treated "as is" in this configuration; in the case of fibers, however, the fibers may be formed into a fabric prior to being treated.

**(Working Examples)**

Next, the present invention will be described more concretely in terms of working examples.

**Working Examples 1 and 2 and Comparative Examples 1 Through 4**

Two liquid crystal polyesters (referred to hereinbelow as "polymers A and B") consisting of polyethylene terephthalate and p-acetoxybenzoic acid were manufactured, and these polyesters were formed into films by the inflation method, thus producing biaxially oriented films with a thickness of 40 μm.

Polymer A: Liquid crystal polyester consisting of a p-hydroxybenzoic acid component (75 mol%) and a polyethylene terephthalate component (25 mol%).

Polymer B: Liquid crystal polyester consisting of a p-hydroxybenzoic acid component (80 mol%) and a polyethylene terephthalate component (20 mol%).

The films thus obtained were subjected to a roll calender treatment (working examples) or a hot pressing treatment (comparative examples).

(1) Roll Calender Treatment

A treatment was performed at a line pressure of 300 kg/cm, a roll temperature of 160°C and a roll circumferential speed of 3 m/min using a MagnaPower roll calender manufactured by Bellmatic.

(2) Hot Pressing Treatment

A treatment was performed for a pressing time of 30 minutes at a pressure of 50 kg/cm$^2$, a temperature of 160°C (Comparative Examples 1 and 3) or for a pressing time of 1 second at a pressure of 1 kg/cm$^2$ and a temperature of 270°C using a pressing machine manufactured by Shinto Metal (Comparative Examples 2 and 4).

The films before and after the abovementioned treatments were rubbed with steel wool #0000 at a pressing pressure of 10 kg/cm$^2$, and the conditions of interlayer peeling of the films were observed. Furthermore, the tensile strength of the films before and after treatment was measured.

The results obtained are shown in Table 1.

Table 1

| | Polymer | Treatment method | Film before treatment | | Film after treatment | |
|---|---|---|---|---|---|---|
| | | | Strength (MD/TD) | Peeling | Strength (MD/TD) | Peeling |
| Comparative Example 1 | A | (2) | 30/21 | Frequent | 30/21 | Frequent |
| Comparative Example 2 | " | " | " | " | -- | None |
| Working Example 1 | " | (1) | " | " | 28/21 | " |
| Comparative Example 3 | B | (2) | 26/21 | " | 26/21 | Frequent |
| Comparative Example 4 | " | " | " | " | -- | None |
| Working Example 2 | " | (1) | " | " | 25/19 | " |

Notes
(1) The units of strength (tensile strength) are kg/mm$^2$
(2) In Comparative Examples 2 and 4, the film deteriorated as a result of treatment, so that measurement of the strength was impossible.

**(Effect of the Invention)**

The present invention makes it possible to obtain molded articles that are free from any problems of fibrillation or interlayer peeling without losing the superior mechanical characteristics of liquid crystal polymer molded articles.

Applicant: Unitika, Ltd.